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DETERMINATION OF ELEVATED-TEMPERATURE FATIGUE DATA
ON REFRACTORY ALLOYS IN ULTRA-HIGH VACUUM

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Prepared for:

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
CONTRACT NO. NAS 3-6010

TECHNICAL MANAGEMENT

Paul E. Moorhead
Space Power Systems Division
NASA - Lewis Research Center

January 15, 1966

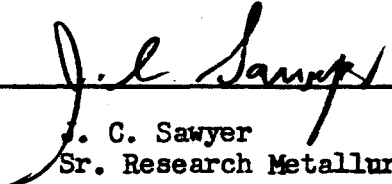
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FOREWORD

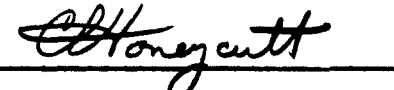
The work described herein is being performed by TRW Inc. under the sponsorship of the National Aeronautics and Space Administration under Contract NAS 3-6010. The purpose of this study is to obtain fatigue life data on refractory metal alloys for use in designing space power systems.

The program is administered for TRW Inc. by E. A. Steigerwald, Program Manager, C. R. Honeycutt and J. C. Sawyer are the Principal Investigators. The NASA technical director is P. E. Moorhead.


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I INTRODUCTION

The purpose of this investigation is to generate fatigue data for refractory alloys at elevated temperatures in ultra-high vacuum environments. The ultimate objective is to determine whether fatigue life or creep is the limiting design parameter in turbine applications involving space-power systems.

Previous reports have described the design and construction of equipment for conducting fatigue tests in vacuum chambers at temperatures up to 3000°F. The application of the cyclic stress is accomplished by a piezoelectric system operating at approximately 20 KHz. Using this apparatus fatigue tests have been conducted on notched specimens ($K_T = 1.75$) of TZM at 1800°F (982°C) and TZC at 2000°F (1093°C).

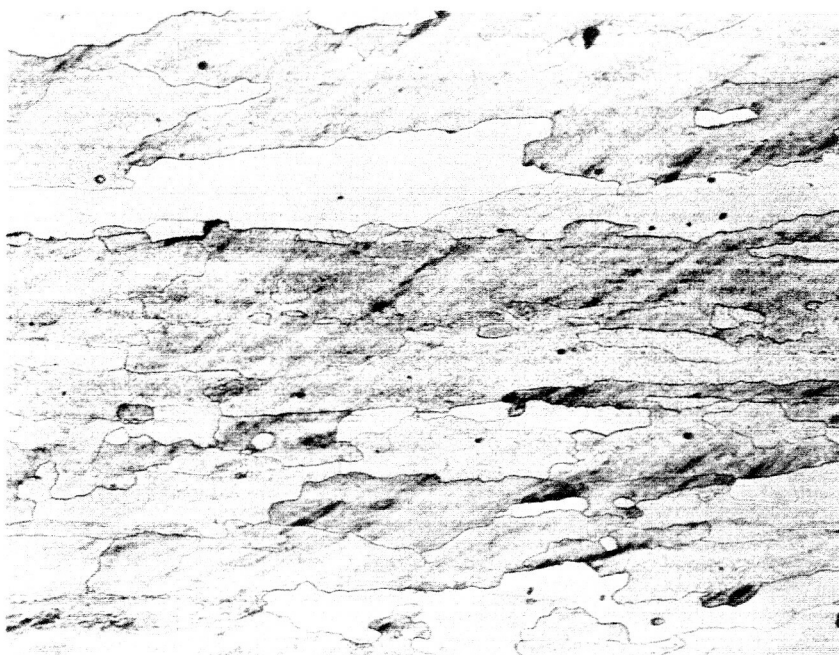
During this report period additional notched fatigue tests were conducted at 2000°F (1093°C) on TZC alloy from a second heat of material. The fatigue curve was extremely sensitive to applied stress and a decrease in peak dynamic stress values from 20.1 Ksi (1.38×10^8 N/m²) to 15.6 Ksi (1.07×10^8 N/m²) produced a variation in failure time for 0.6 hours (4.1×10^7 cycles) to greater than 143 hours ($> 9.86 \times 10^9$ cycles). Tests conducted on smooth specimens of TZC bar stock defined the resonant wave length at 2000°F (1093°C), however, no failure occurred in this material at a peak dynamic stress of 23.9 Ksi (1.65×10^8 N/m²) after 500 hours (3.6×10^{10} cycles).

II MATERIALS

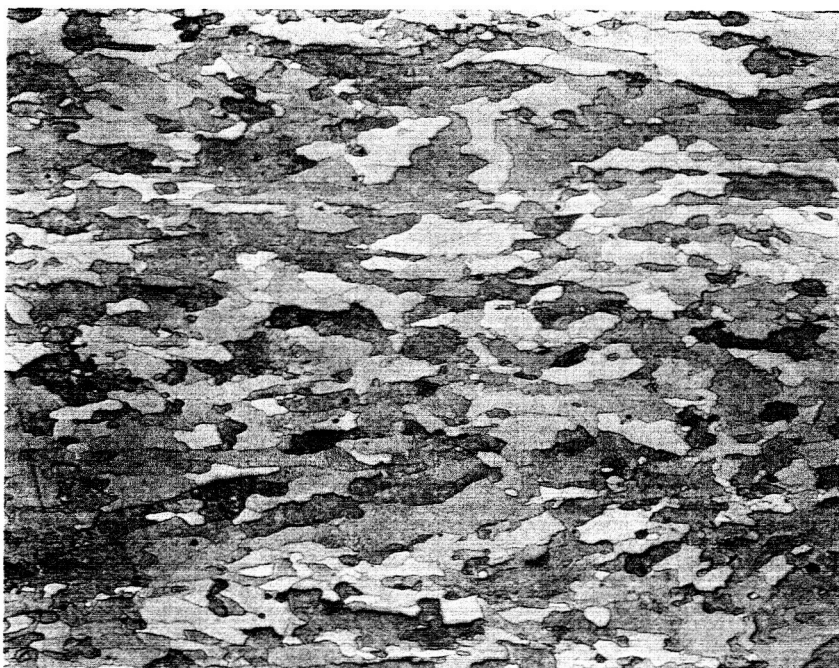
The molybdenum-base alloys, TZM and TZC have been included in the test program. The specific material form, heat numbers, and composition are presented in Table 1. The TZM alloy has been employed primarily as a working material to define the effects of notch geometry and to optimize the design of the load train assembly. The TZC alloy has been evaluated as bar stock and plate material from two heats (M-89 and M-91). The processing history of the TZC plate, described in the fifth quarterly report, CR 54775, was significantly different for each of the heats being evaluated. All the tests conducted to date on the TZM were performed with the material in the stress-relieved condition. With the exception of one test which was performed on the stress-relieved material, the TZC specimens were annealed at 3092°F (1700°C) for one hour in vacuum prior to testing. Typical microstructures of the TZC from Heats M-89 and M-91 after annealing are shown in Figure 1. Some cracking occurred in the TZC specimens from Heat M-91 during heat treatment presumably as a result of the fact this heat was rolled at a lower temperature and, therefore, had a higher degree of residual stress. Varying the heating and cooling rate between 3200°F/hr. and 1600°F/hr. did not alter the tendency for cracking in the machined specimens. When the annealing treatment was performed on the as-received plate, only slight surface fissures were evident and sufficient stock was available to allow full-size specimens to be machined from the plate.

TABLE 1
CHEMICAL COMPOSITION OF ALLOYS TESTED

<u>Material</u>	<u>Form</u>	<u>Vendor</u>	<u>Heat</u>	<u>Mo</u>	<u>Composition - Wght. %</u>			<u>Condition</u>
					<u>Zr</u>	<u>Ti</u>	<u>C</u>	
TZM	1/2" dia. Bar	Climax	7463	Bal.	0.08	0.48	0.016	Stress Relieve, 1 hour at 1232°C
TZM	1/2" dia. Bar	Climax	7468	Bal.	0.09	0.50	0.022	Stress Relieve, 1/2 hour at 1232°C
TZC	1/2" dia. Bar	Climax	4263	Bal.	0.38	1.24	0.10	Stress Relieve 1/2 hour at 1204°C
TZC	1/2" dia. Bar	Climax	4230-2	Bal.	0.29	1.35	0.089	Stress Relieve 1/2 hour at 1315°C or anneal 1 hour at 1700°C
TZC	0.60" Plate	G. E.	M-89	Bal.	0.20	1.45	0.13	Anneal at TRW 1700°C, 1 hour ASTM Grain Size 1 x 4
TZC	0.60" Plate	G. E.	M-91	Bal.	0.18	1.25	0.14	Anneal at TRW, 1700°C, 1 hour ASTM Grain Size 4 x 6



Heat M-89



Heat M-91

Figure 1. Microstructure of TZC Material, Annealed 3092°F (1700°C), 1 Hour, Etchant: 15%HF, 15% H₂SO₄, 8%HNO₃, 62%H₂O, 100X.

III PROCEDURE

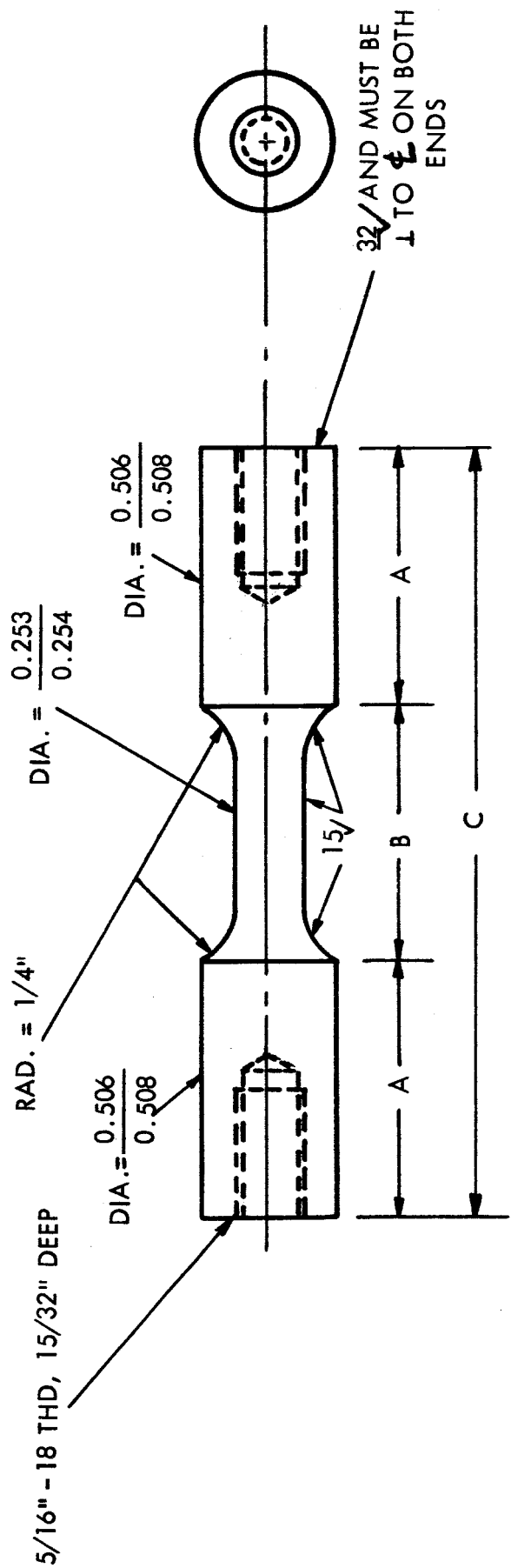
The program plan involved conducting fatigue tests at approximately 20 kHz on both smooth and notched specimens. Since sufficient stress could not be generated on the smooth specimens to consistently produce a failure at a test temperature of 2000°F (1093°C) considerable effort was devoted to varying the load train design so that increased ultrasonic drive could be developed.

The test specimen geometries are shown in Figures 2, 3, and 4. Two types of smooth specimens were used. The initial design consisted of a dumb-bell type and was selected on the basis of calculations presented by Neppiras^{1*}. During the course of the testing it became evident that the specimen dimensions required alteration to develop resonant conditions at the test temperature and the optimum drive frequency for the ultrasonic generator. This initial specimen tuning was more readily accomplished with the stepped specimen shown in Figure 3. The notch specimen (Figure 4) was a dumb-bell type with a 1/4" major diameter, a 1/32" notch radius and a minor diameter of 0.174". The test method involved mechanically mounting the specimen to the drive train, pumping the units to a vacuum better than 1×10^{-8} Torr at room temperature, and then heating the specimen at a rate so that the pressure never exceeded 1×10^{-6} Torr.

A W-3% Re/W-25% Re thermocouple placed approximately 1/8 inch from the surface at the specimen midpoint was used for temperature measurement. Due to breakage produced by the vibration, the thermocouple could not be attached directly to the specimen. The temperature was stabilized for approximately two hours and then the cyclic load was applied.

As a result of the application of the high frequency cyclic load, heating of the fatigue specimen took place. The degree of heating was dependent upon the power applied to the system. In determining the S-N curve, the ambient test temperature; i.e., the temperature recorded by the thermocouple, was set at a fixed value for each test. At the high stress levels where significant heating of the specimen occurred, the test time was sufficiently short so that a readjustment of the furnace temperature to compensate for the self-heating could not be accomplished. At the low values of applied dynamic stress, the temperature increase was very slight and no adjustment of the furnace temperature was usually necessary. Although the data are presented for constant values of the ambient temperature, the actual specimen temperature was also recorded in cases where the test duration was sufficient to allow time for accurate readings. The temperature increase due to self-heating was obtained by measuring the difference in specimen brightness temperature before and after the application of the cyclic load with an L-N optical pyrometer.

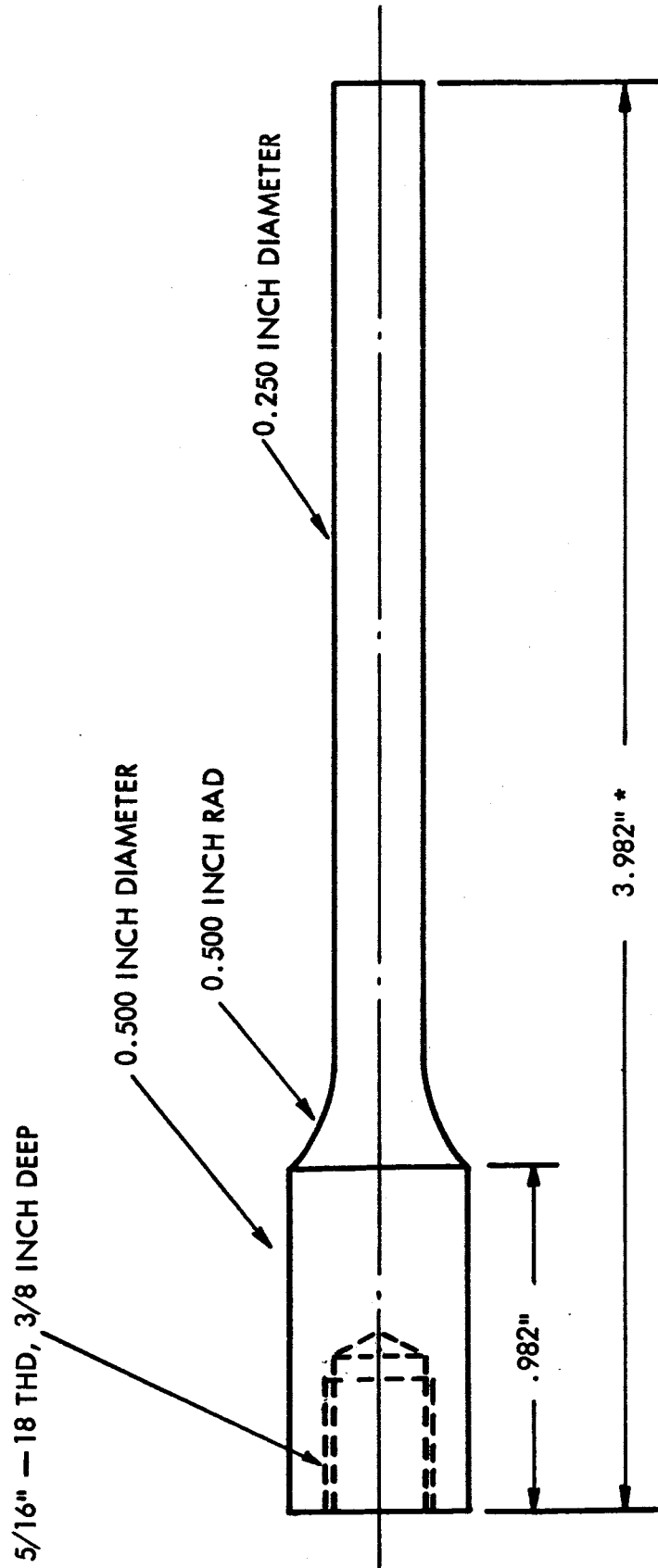
* Superscript numbers pertain to references in the Bibliography.



ALL DIAMETER & THD. ENDS MUST BE CONCENTRIC ± 0.002 T.I.R.
USE MINIMUM RELIEF ON THD. ENDS.

	A (IN.)	B (IN.)
TZC	0.980	0.980
TZM	1.155	1.155

FIGURE 2 DUMB-BELL TYPE SMOOTH SPECIMEN GEOMETRY



*LENGTH ADJUSTED FOR RESONANCE WITH DRIVE TRAIN AT TEST TEMPERATURE

FIGURE 3 GEOMETRY OF STEPPED FATIGUE SPECIMEN

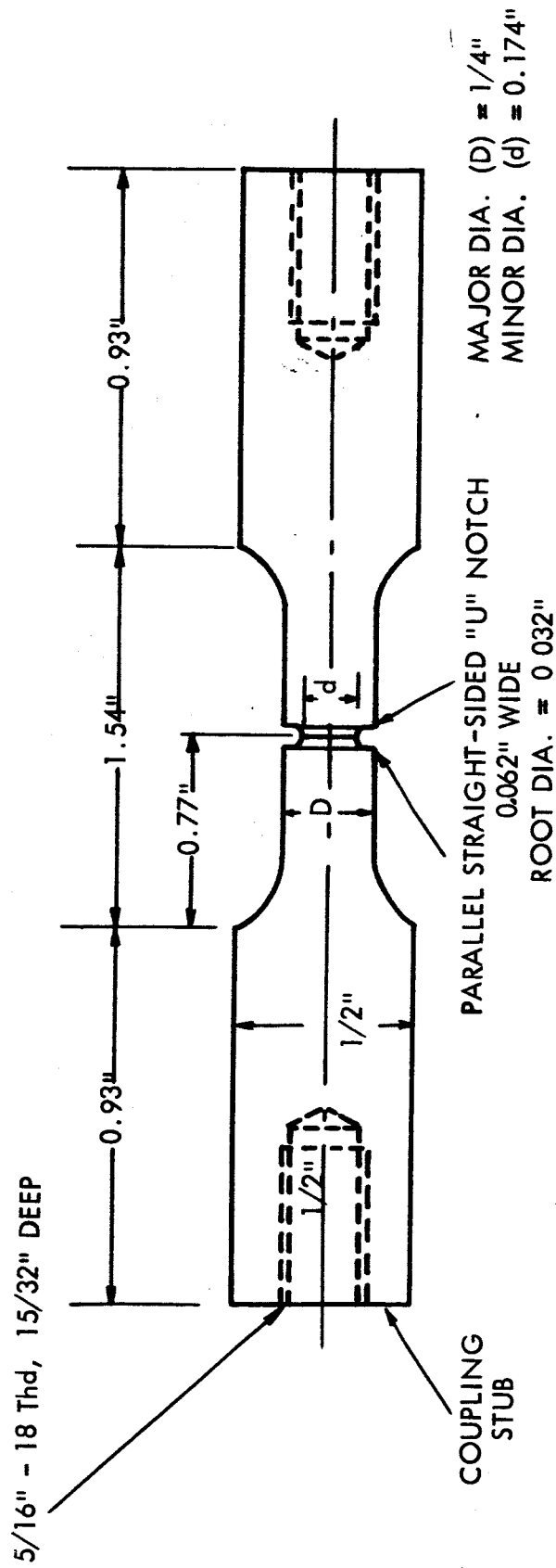


FIGURE 4 GEOMETRY OF NOTCH FATIGUE SPECIMEN

The applied dynamic stress produced by the ultrasonic vibration was determined from displacement measurements made directly on the specimen with a cathetometer. In the dumb-bell specimens two reference points were selected approximately equidistant from the specimen midpoint and the displacements at these points were determined by averaging 30 readings. The variation in these readings was approximately $\pm 50 \mu$ -in. The displacement along the specimen was assumed to follow the sinusoidal relationship:

$$\delta_x = \delta_o \sin \frac{2\pi x}{\lambda} \quad (1)$$

where: $2\delta_x$ is the total measured displacement at a point X from the specimen midpoint,
 δ_o is the maximum amplitude, and
 λ is the resonant wave length.

The maximum strain at the midpoint of the dumb-bell type specimen can then be determined from the equation:

$$\epsilon_{\max} = \frac{2\pi \delta_o}{\lambda} \quad (2)$$

and the dynamic stress (σ) is the product of the strain and the elastic modulus at the particular test temperature

$$\sigma = (\epsilon_{\max}) (E) \quad (3)$$

In the case of the stepped specimen the stress was determined by measuring the displacement of the specimen end ($2\delta_o$) which was used with equations (2) and (3) to provide a stress value.

When the notch specimen was employed, the calculations of stress based on the displacement measurements taken on the major diameter must be increased by the area ratio 2.11 and the theoretical stress concentration factor* ($K_T = 1.75$). On this basis the stress in the notch specimen was calculated from the following relation:

$$\sigma = 3.69 \left(\frac{2\pi}{\lambda} \delta_o E \right) \quad (4)$$

* Previous reports on this program used an effective stress concentration factor $K_f = 1.5$ rather than the theoretical stress concentration factor (K_T) of 1.75. In the current report the theoretical factor will be used since this represents the maximum factor and since some doubt still exists as to the exact value of K_f which is applicable.

All the tests were conducted in the 18.0 to 21.0 kHz range. Cracking of the test specimen was accompanied by a significant decrease in the resonant frequency. Although the end point was defined as the point when this rapid shift in resonance occurred the test was continued until the desired amplitude could no longer be obtained. This condition usually resulted in propagating the fatigue crack through approximately one-half the specimen cross-section.

IV RESULTS AND DISCUSSION

1. Smooth Specimen Tests

The test results obtained from the smooth specimen geometry are summarized in Table 2. The initial tests were conducted with stepped specimens of TZC bar stock. The purpose of the first test performed on TZC (stress relieved 1/2 hour at 2200°F, 1204°C) was to determine the resonant wave length of the material at the 2000°F (1093°C) test temperature. Premature failure occurred in the specimen at a very slight tool mark after 24 minutes of operation (2.9×10^7 cycles) at a peak stress of 26.8 Ksi ($1.85 \times 10^8 \text{ N/m}^2$). A second test was performed on TZC bar stock using a ground specimen (surface finish $< 15 \text{ RMS}$) that was annealed at 3092°F (1700°C) for 1 hour. In this case the applied stress level was decreased to 23.9 Ksi ($1.65 \times 10^8 \text{ N/m}^2$) in an effort to obtain a longer test period which would allow measurements of displacement to be performed along the length of the rod. The variation of displacement along the rod is shown in Figure 5. The results indicate that the displacement in the TZC at the ambient temperature of 2000°F (1093°C) follows a cosine function and has a resonant wave length of 9.80 inches. Using this value of wave length, the density from room temperature measurements and the coefficient of expansion, the elastic modulus at 2000°F (1093°C) was determined to be 35.8×10^6 psi which is in excellent agreement with the published values for the molybdenum alloy at the same temperature².

The test on the stepped specimen at a peak stress of 23.9 Ksi ($1.65 \times 10^8 \text{ N/m}^2$) and 2000°F (1093°C) was continued for 500 hours (3.6×10^{10} cycles) without producing any indication of failure. After this test period the drive voltage applied to the specimen was increased to produce a stress of 30 Ksi ($2.07 \times 10^8 \text{ N/m}^2$) and the test was continued for 16 hours (1.15×10^9 cycles) without producing a failure. Additional tests will be performed with the stepped specimen to determine the maximum dynamic stress that can be generated in the apparatus. Using the measured resonant wave length of 9.80 inches, the smooth dumb-bell type specimens were redesigned to a total length of 2.94 inches and tests will be conducted with this configuration at an A* ratio of 00, 0.67, and 0.25.

2. Notch Specimen Tests

The notch fatigue tests during this quarter were performed at 2000°F (1093°C) on TZC material from Heat M-91, annealed 1 hour at 3092°F (1700°C). A summary of the test results are presented in Table 3 and Figure 6. As in the previously reported results for the stress relieved TZM, the notch fatigue properties of the recrystallized TZC were extremely sensitive to applied stress.

* Ratio of dynamic to mean stress

TABLE 2

SUMMARY OF SMOOTH FATIGUE TESTS ON TZC, AMBIENT TEMPERATURE 2000°F (1093°C)

VACUUM ENVIRONMENT $< 1 \times 10^{-7}$ TORR, 20 KHZ

Specimen Type	Material Form	Amplitude (δ_o) At Specimen End (μ -in.)	Strain (Em) in/in	Stress Based On A Modulus of 35.8×10^6 psi (Ksi, 6.89×10^6 N/m ²)	Failure Time (Hours)	Number of Cycles To Failure
Stepped	Bar, SR, 1/2 hour at 1204°C, Heat 7463	1170	0.748×10^{-3}	26.8	0.40 *	2.9×10^7
Stepped	Bar, annealed, 1 hour 1700°C, Heat 7463	1045	0.668×10^{-3}	23.9	> 500	$> 3.6 \times 10^{10}$
Stepped	Bar, annealed, 1 hour 1700°C, Heat 7463	950	0.609×10^{-3}	21.8	> 503	$> 73.6 \times 10^{10}$

* Failure occurred at a small mark located at the specimen node.

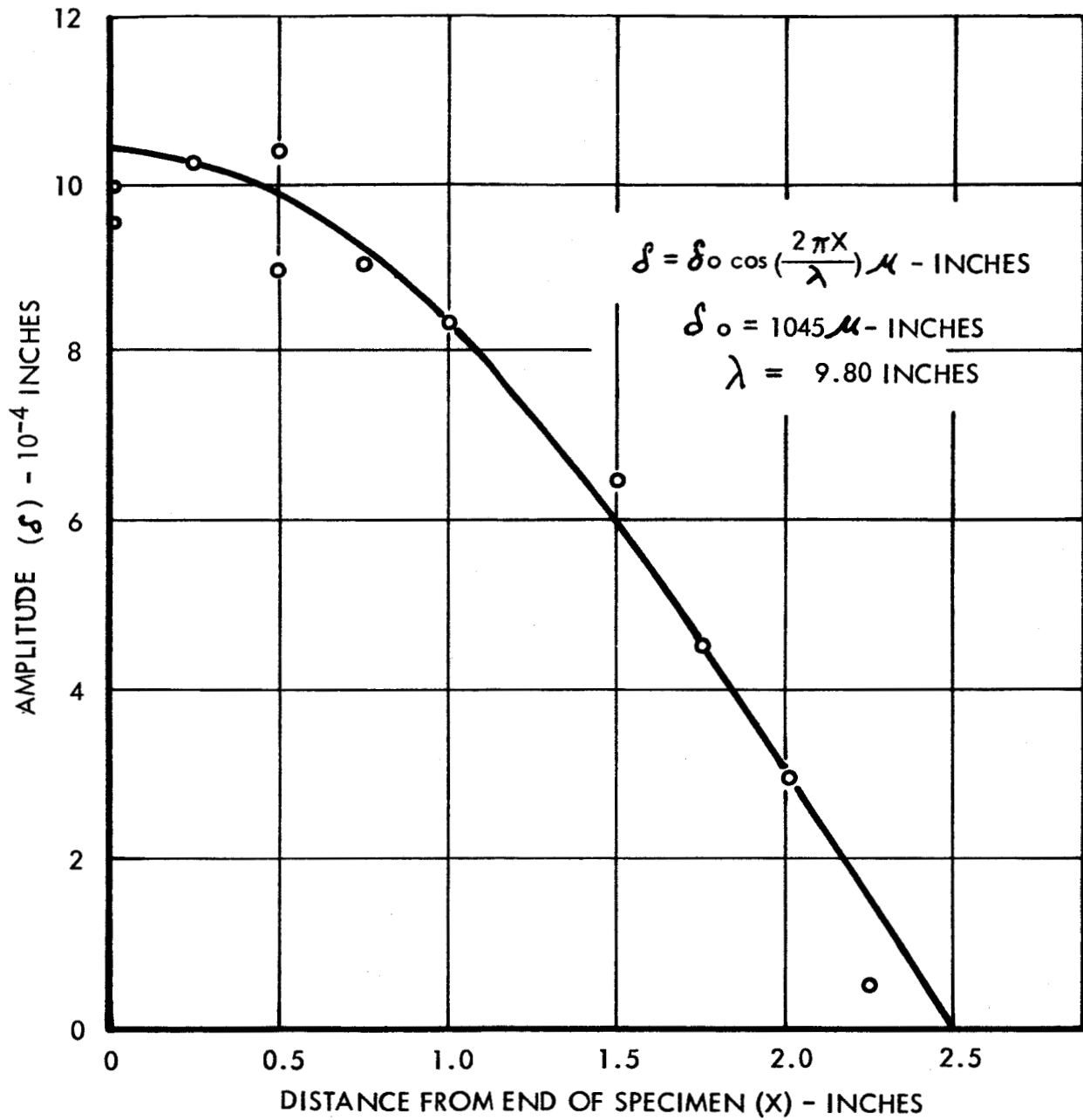


FIGURE 5 AMPLITUDE AS A FUNCTION OF SPECIMEN POSITION, TZC RECRYSTALLIZED AT 3092°F (1700°C), AMBIENT TEST TEMPERATURE 2000°F (1093°C), 20 KCS (kHz).

TABLE 3
SUMMARY OF NOTCH FATIGUE TESTS ON TZC PLATE (HEAT H-91), RECRYSTALLIZED 3092°F (1700°C)
AMBIENT TEST TEMPERATURE 2000°F (1093°C), VACUUM ENVIRONMENT $< 1 \times 10^{-7}$ TORR

Specimen	Strain (μ -in/in)	(A) Dynamic Stress Based On Smooth Specimen Ksi ($6.90 \times 10^6 \text{ N/m}^2$)	(B) Peak Dynamic Stress Ksi ($6.90 \times 10^6 \text{ N/m}^2$)	(C) Static Notch Stress Ksi ($6.90 \times 10^6 \text{ N/m}^2$)	(D) Total Peak Stress Ksi ($6.90 \times 10^6 \text{ N/m}^2$)	Cycles To Failure (KHz)	Time To Failure (Hours)	Temp. Increase Due to Drive °F
17	109	3.90	14.4	1.19	15.6	$> 9.98 \times 10^{10}$	> 14.3	0
17*	114	5.14	18.9	1.19	20.1	4.1×10^7	0.60	-
18	92.7	3.32	12.2	18.4	30.6	6.8×10^6	0.10	-
20	132	4.72	17.4	1.19	18.6	2.6×10^7	0.37	-
22	61.2	2.19	8.05	14.4	22.5	$> 3.1 \times 10^8$	> 4.5	10
26	66.7	2.39	8.88	14.4	23.3	$> 5.5 \times 10^7$	> 0.80	0
27	120	4.29	15.9	1.20	17.1	4.6×10^7	0.67	24
28	125	4.46	16.5	1.20	17.7	5.0×10^7	0.73	18

* Retested after testing at a lower applied stress.

Column A: Calculated by multiplying strain by elastic modulus (35.8×10^6 psi)

Column B: Column A multiplied by $K_t = 1.75$ and ratio of major-to-minor diameter square $(D/d)^2 = 2.11$

Column C: Static average stress (drive train and weights below specimen) multiplied by stress concentration factor $K_t = 1.75$

Column D: Summation of Columns B and C.

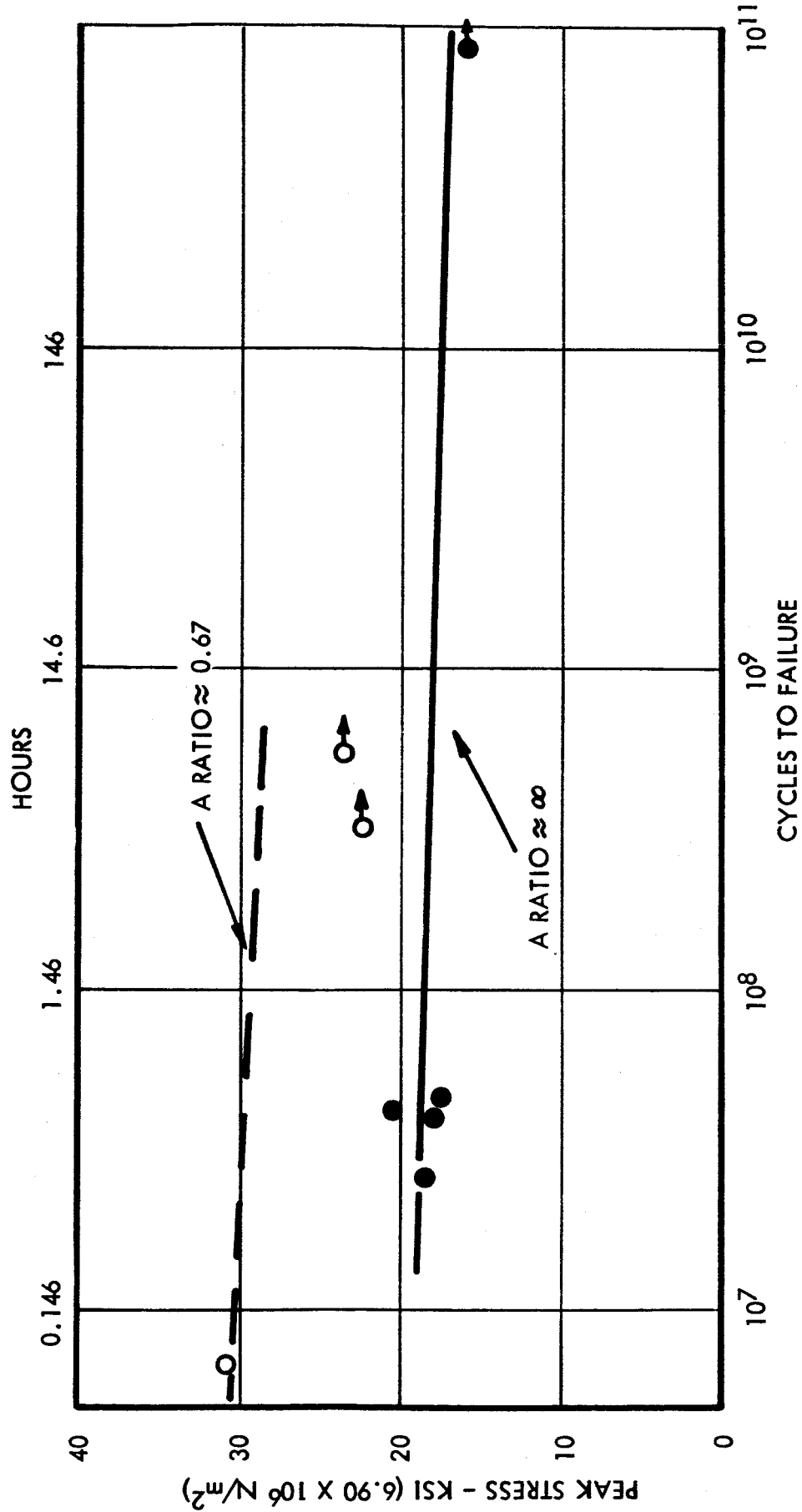


FIGURE 6 FATIGUE CURVES FOR TZC, HEAT M-91, RECRYSTALLIZED AT 3092°F (1700°C) TESTED AT AN AMBIENT TEMPERATURE OF 2000°F (1093°C) IN A VACUUM ENVIRONMENT TEST FREQUENCY 20 kHz.

The points obtained on the TZC at an A ratio of approximately 0.67 are also presented in Figure 6.

A comparison between the notch fatigue test results from TZC Heats M-89 and M-91 and TZM (Heat 7463) is given in Figure 7. In general the fatigue life has the expected very strong dependence on applied stress. An exception to this generalization was evident in three test points obtained from TZC specimens from Heat M-89. No explanation is available for this apparent difference between the two heats of materials at the short test times.

As reported in the Fifth Quarterly Report no reason is apparent as to why the notch specimens of TZC from Heat M-91 failed at a calculated peak stress as low as 10.5 Ksi ($7.24 \times 10^8 \text{ N/m}^2$) while smooth specimens from the same heat did not fail at a peak stress of 15 Ksi ($1.03 \times 10^8 \text{ N/m}^2$) after 69 hours (4.8×10^8 cycles). Since the use of the theoretical stress concentration factor and the area ratio represent the maximum stress elevation, they should tend to overestimate the peak stress value. A question existed as to whether the use of high frequency would produce a heterogeneous stress distribution at the notch which was greater than the simple area ratio correction. To examine the point, room temperature tests were conducted at 20.2 KHz on Armco iron using specimens with varying stress concentration factors. A stepped specimen was used and cooling was accomplished by immersing the specimen in a water bath. A summary of the test data is presented in Table 4 and plotted in terms of calculated peak stress in Figure 8. The results indicate that despite statistical scatter there is relatively good agreement in the data when presented in terms of peak stress values. On this basis the difference between the notch and smooth results does not appear to be related to gross errors in the notch factor calculations. Additional tests with varying notch geometries will be conducted with TZM at 2000°F (1093°C).

3. Equipment Modification

In an effort to increase the displacement produced by the ultrasonic drive system several modifications were made in the design of the amplification horns. A comparison between the initial design and the resultant modification is schematically shown in Figure 9. The modification involved the following factors:

1. Eliminating the thick weld joint at the vacuum chamber flange,
2. Substituting a 1/2" diameter stub for the 1" stub as the primary train member, and
3. Boring out the center of the stubs.

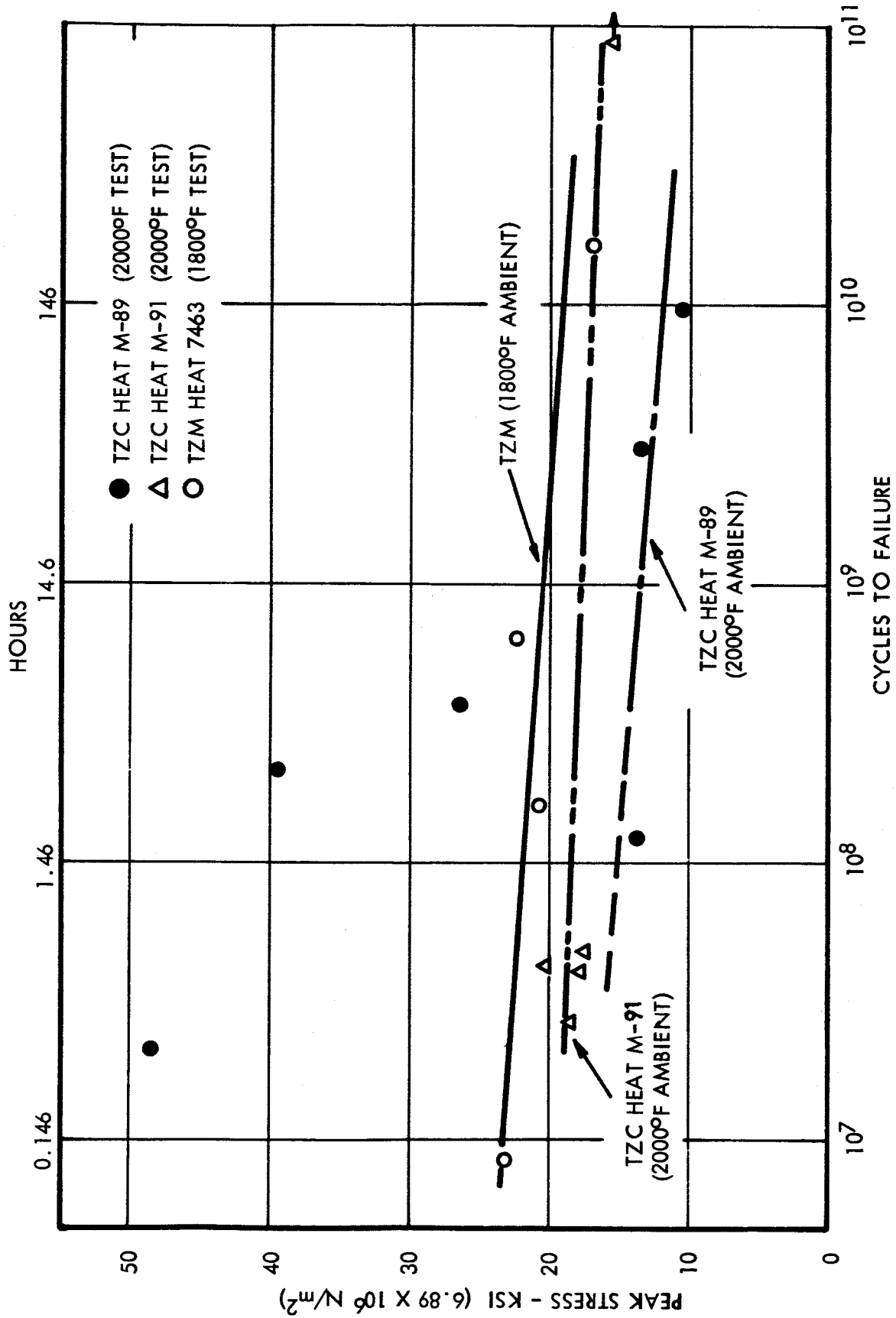


FIGURE 7 FATIGUE CURVES FOR TZC (RECRYSTALLIZED AT 3092°F) AND TZM (STRESS RELIEVED CONDITION) TESTED IN VACUUM ENVIRONMENT $< 10^{-8}$ TORR.

TABLE 4

SUMMARY OF NOTCH FATIGUE DATA ON ARMCO IRON, TESTED WITH VARIOUS STRESS CONCENTRATION

FACTORS AT ROOM TEMPERATURE, WATER COOLANT, TEST FREQUENCY, 20KHZ A = ∞

Specimen	(A)		(B)		(C)		(D)		Cycle to Failure
	Smooth Stress Ksi ($6.90 \times 10^6 \text{ N/m}^2$)	Smooth Stress Ksi ($6.90 \times 10^6 \text{ N/m}^2$)	Stress Concentration Factor K_T	Stress Concentration Factor K_T	Area Ratio (D/d) ²	Area Ratio (D/d) ²	Peak Stress Ksi ($6.90 \times 10^6 \text{ N/m}^2$)	Peak Stress Ksi ($6.90 \times 10^6 \text{ N/m}^2$)	
1	34.7	34.7	1.0	1.0	1.0	1.0	34.7	34.7	3.12×10^7
2	37.1	37.1	1.0	1.0	1.0	1.0	37.1	37.1	1.08×10^7
3	32.2	32.2	1.0	1.0	1.0	1.0	32.2	32.2	6.62×10^5
4	~29.7	~29.7	1.81	1.81	1.13	1.13	61.0	61.0	~ 3.74×10^7
5	17.9	17.9	1.80	1.80	1.25	1.25	40.4	40.4	1.53×10^7
6	11.9	11.9	1.85	1.85	1.24	1.24	27.3	27.3	4.74×10^7
7	9.9	9.9	1.92	1.92	1.67	1.67	31.6	31.6	4.18×10^5
8	~19.8	~19.8	2.03	2.03	1.64	1.64	65.9	65.9	~ 3.74×10^6
9	14.9	14.9	2.04	2.04	1.60	1.60	48.6	48.6	8.79×10^5
11	~9.9	~9.9	1.76	1.76	3.50	3.50	61.2	61.2	~ 9.38×10^5
12	~19.8	~19.8	1.88	1.88	3.40	3.40	126.5	126.5	~ 2.50×10^6
13	5.6	5.6	1.68	1.68	3.57	3.57	60.0	60.0	4.76×10^6
14	39.6	39.6	1.0	1.0	1.0	1.0	39.6	39.6	2.3×10^7

Column D is obtained by multiplying columns A, B, and C.

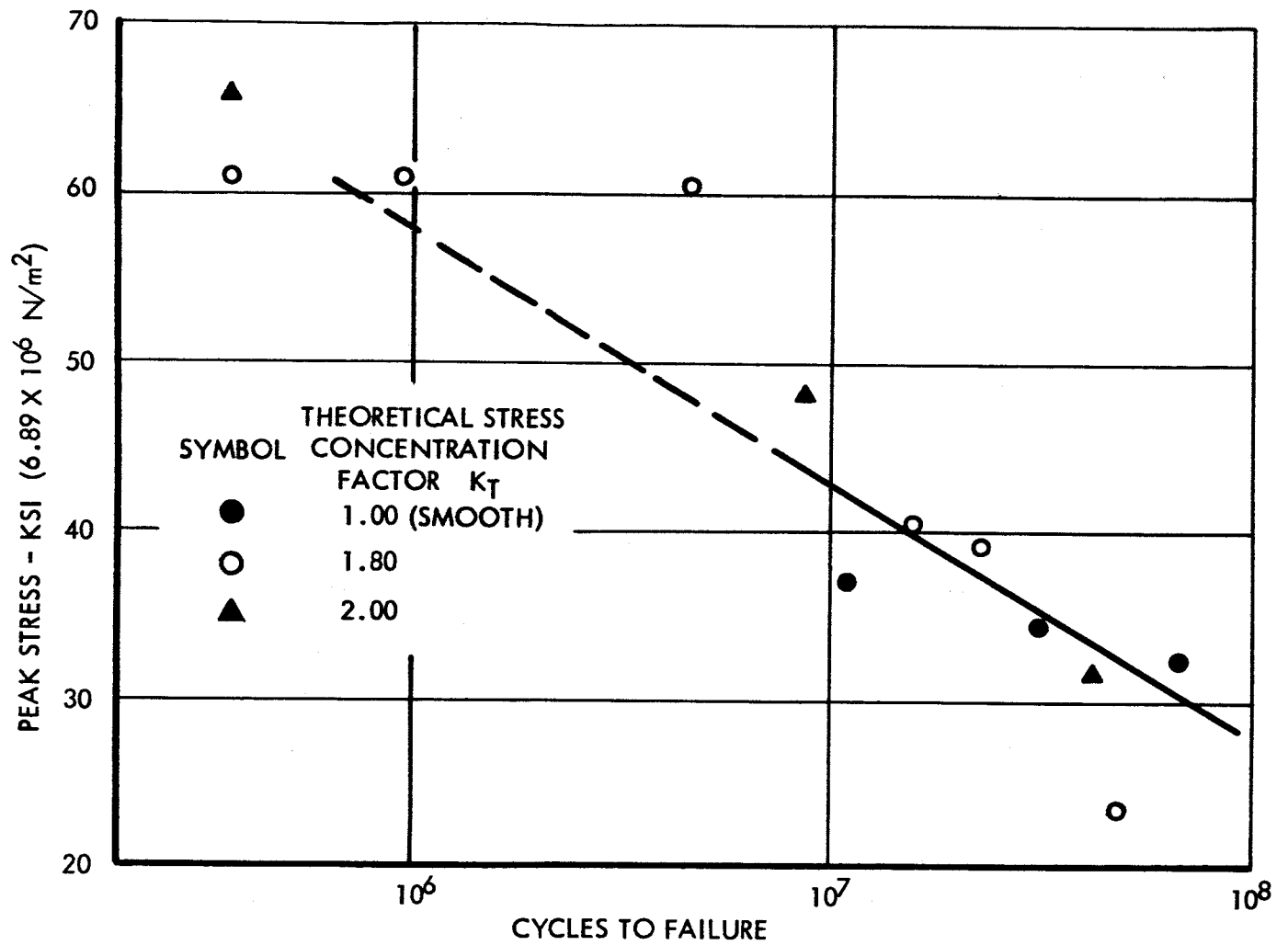


FIGURE 8 FATIGUE CURVE FOR ARMCO IRON TESTED AT ROOM TEMPERATURE, 20 kHz WITH VARIOUS NOTCH CONCENTRATION FACTORS

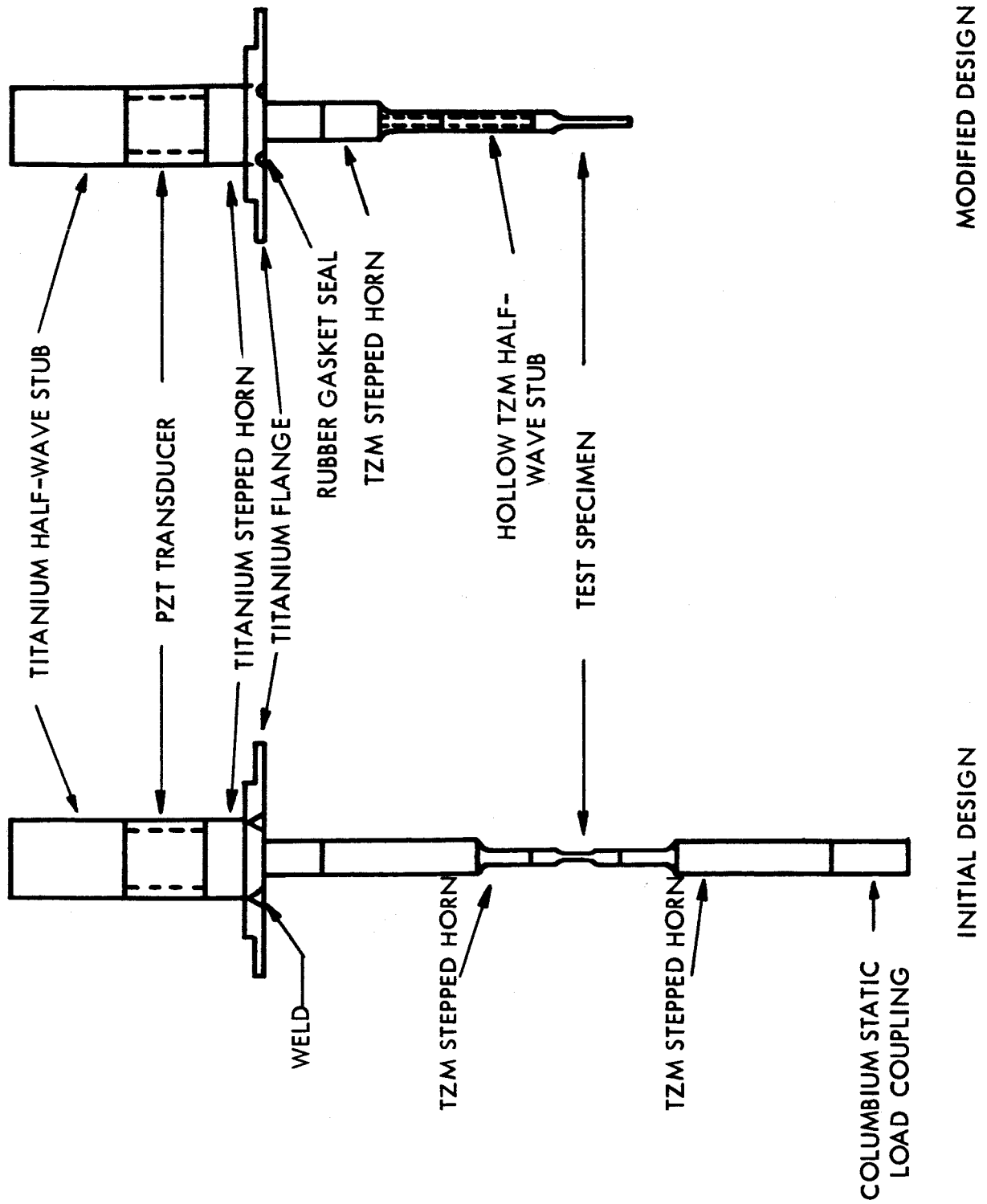


FIGURE 9 VARIATIONS IN DESIGN OF ULTRASONIC DRIVE TRAIN

The removal of the thick weld joint eliminated a possible constraint at the flange. The Viton seal which is currently being used, will ultimately be replaced by a thin welded or brazed diaphragm. The use of a hollow 1/2" stub in place of the 1" diameter stub as the primary train component reduced the mass that the drive system must vibrate if operating slightly off resonance. Each individual component of the drive train was experimentally tuned to insure maximum output.

Attempts were made to further increase the drive output by using the horn design described by Girard and Vidal³. In this design the horn is driven slightly off the nodal point position rather than at the anti-node. Substantial increases in drive amplitude were obtained with this system, however, the sensitivity to tuning was very critical. Due to this tuning sensitivity the use of the modified horn design was discontinued.

A second approach to increase the drive output involved obtaining a commercially-available 1000 watt magnetostrictive* unit. Although this unit is still in the process of being evaluated, displacements greater than 4000 μ -inches have been measured at the end of the stepped specimens when these specimens are maintained at room temperature. When cooling is eliminated so that self-heating occurs or when the specimens are heated in the vacuum test chambers, a substantial decrease in output occurs despite the fact that the system is satisfactorily tuned for the particular test temperature. Typical results obtained on the magnetostrictive drive systems at various test temperatures are shown in Table 5. These results coupled with previous data on the piezoelectric system indicate that the higher test temperature may be producing a substantial increase in damping which minimizes the resulting displacements. Tests will be conducted with the magnetostrictive drive at high power inputs in an attempt to determine the extent to which damping minimizes the available drive output.

* Blackstone Corporation, Sheffield, Pennsylvania

TABLE 5

INFLUENCE OF TEST TEMPERATURE ON THE DRIVE AMPLITUDE

<u>Drive System</u>	<u>Ambient Temperature</u>		<u>Double Amplitude ($2\delta_o$)</u> <u>(μ- inches)</u>	<u>Resonant Frequency (KHZ)</u>
	<u>°F</u>	<u>°C</u>		
Piezoelectric	76	24	1800	20.65
"	1100	593	1100	20.30
"	2000	1093	900	19.92
Magnetostrictive	76	24	2500	22.02
"	1200	593	2000	20.98
"	2000	1093	1500	20.39

V FUTURE WORK

Notch tests will be conducted on TZC plate material to produce fatigue curves at A ratios of ∞ to 0.67. Tests on the smooth specimen geometry will be continued at an A ratio of 0.67 and 0.25 in an attempt to produce a meaningful Goodman-type diagram. The influence of notch geometry on the fatigue strength of the molybdenum alloys at elevated temperatures will be studied in TZM bar stock in an attempt to define the reason for the differences between smooth and notch test results.

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